PREPARATION AND CHARACTERIZATION OF RECYCLED CARBON FIBER / CARBON NANOTUBE REINFORCED EPOXY COMPOSITES

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A thesis submitted

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DECLARATION

I declare that this thesis entitled "Preparation and Characterization of Recycled Carbon Fiber / Carbon Nanotube Reinforced Epoxy Composites" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : .....

Name : .....

Date : .....

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature :…….....

Supervisor Name :…….....

Date :…….....

DEDICATION

To my beloved parents and brothers

ABSTRACT

The usage of carbon fiber reinforced polymers (CFRP) has been growing at a substantial rate that leads to the increasing amount of waste generated from end-of-life components and manufacturing scrap. Recognizing the hazardous waste that would be harmful to the human health as well as the environment, this has poses the need to recycle the waste. This report presents the research of compression and wear behaviour of as-received as well as treated rCF and carbon nanotube (CNT) reinforced epoxy composites. The rCF derived from mechanically recycled CFRP composite woven prepreg waste - materials impregnated with epoxy resin is of low value commodity. Therefore, cyclic cryogenic treatment is introduced to produce valuable product of rCF. The objectives of this study were to investigate the effect of cyclic cryogenic treatment on the recycled carbon fiber as well as to study the performance of the as-received rCF reinforced epoxy (EP/rCF-AR), treated rCF reinforced epoxy (EP/rCF-T) and carbon nanotube reinforced treated rCF and epoxy (EP/rCF-T/CNT) composites. The composites' samples were fabricated using vacuum casting technique. The samples were then subjected to physical, compression and tribological testing. In addition, microscopy examinations were carried out to observe and to analyse the morphology of the worn surfaces. It was found that at 25 cycles of cryogenic treatment, the epoxy resin on the surface of rCF is nearly absent, indicating the effective removal of epoxy resin due to the mismatches in thermal expansion between the interface bonding of rCF/epoxy. Based on the compression and wear test analysis, it was observed that the reinforcement effect of rCF-T has improved the compressive strength (4.09 %) and significantly enhanced the wear resistance (32.2 % at 1.0 m/s and 16 N) of epoxy composites as compared to rCF-AR which may attribute to the improved adhesion between the treated rCFs and epoxy matrix. Moreover, further improvement can be seen in EP/rCF-T/CNT as compared to EP/rCF-T for compressive strength (6.18 %) and wear resistance (16.4 % at 1.0 m/s and 16 N). This is because the addition of nano-size CNT in the composite has enhanced the local stress transferred by the matrix and the good reinforcing capability of CNT promotes better adhesion at the polymer matrix interface. From the study, it was clearly shown that the treated rCF was effective in improving the properties of the epoxy composites. Therefore, the treated rCF are valuable product worth to be considered as reinforcements in the composite materials. In addition, nanofillers serve as matrix reinforcement even at low filler content.

ABSTRAK

Penggunaan 'carbon fiber reinforced polymer' (CFRP) yang semakin berkembang banyak telah membawa kepada peningkatan jumlah bahan buangan yang dihasilkan daripada akhir hayat komponen dan hasil buangan semasa pembuatan. Menyedari bahawa bahan buangan tersebut berbahaya kepada kesihatan manusia dan alam sekitar, ia telah menimbulkan kesedaran untuk mengitar semula bahan buangan tersebut. Laporan ini adalah berdasarkan penyelidikan mengenai perlakuan daya mampatan dan daya haus komposit polimer iaitu karbon fiber yang dikitar semula (rCF) dan karbon nanotube (CNT) bertetulangkan epoksi. Karbon fiber yang dikitar semula (rCF) yang digunakan adalah daripada bahan buangan komposit CFRP yang bernilai rendah. Oleh itu, rawatan kitaran cryogenic diperkenalkan untuk menghasilkan produk rCF yang bermutu. Objektif-objektif kajian ini adalah untuk mengkaji kesan rawatan kitaran cryogenic terhadap rCF dan juga mempelajari prestasi rCF yang tidak dirawat bertetulangkan epoksi (EP/rCF-AR), rCF yang dirawat bertetulangkan epoksi (EP/rCF-T) serta rCF yang dirawat dan karbon nanotube (CNT) bertetulangkan epoksi (EP/rCF-T/CNT) komposit. Dalam kajian ini, spesimen komposit dihasilkan melalui teknik 'vacuum casting'. Komposit yang dihasilkan diuji dengan ujian fizikal, ujian mampatan dan ujian tribologi. Di samping itu, mikroskopi digunakan untuk memerhati serta menganalisa morfologi permukaan spesimen yang telah haus. Kajian mendapati bahawa pada kitaran 25 rawatan 'cryogenic', epoksi pada rCF hampir disingkirkan. Hal ini menunjukkan keberkesanan rawatan 'cryogenic' untuk menyingkirkan epoksi oleh rawatan 'cryogenic' yang disebabkan oleh ketidakpadanan pengembangan haba ikatan antara muka rCF dan epoksi. Berdasarkan analisis ujian mampatan dan perlakuan haus, rCF-T menunjukkan peningkatan kekuatan mampatan (4.09 %) serta peningkatan yang ketara dalam rintangan perlakuan haus (32.2 % pada 1.0 m/s dan 16 N) berbanding dengan rCF-AR. Hal ini adalah disebabkan oleh ikatan yang baik antara rCF yang dirawat dengan matrik epoksi. Selain itu, EP/rCF-T/CNT juga menunjukkan peningkatan berbanding EP/rCF-T dalam kekuatan mampatan (6.18 %) dan rintangan haus (16.4 % pada 1.0 m/s dan 16 N). Hal ini adalah disebabkan penambahan saiz nano CNT dalam komposit telah meningkatkan penampungan tekanan yang dipindahkan oleh matriks dan keupayaan pengukuhan CNT telah menggalakkan daya lekatan yang lebih baik di antara muka matriks polimer. Daripada kajian ini, rCF yang dirawat jelas menunjukkan keberkesanan dalam meningkatkan sifat-sifat komposit epoksi. Justeru, rCF yang dirawat adalah produk yang bermutu sebagai pengisi dalam komposit. Tambahan lagi, dapat dilihat juga bahawa penambahan nanopartikel dapat berfungsi sebagai penguat tetulang matriks walaupun pada kandungan pengisi yang rendah.

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LIST OF ABBREVIATIONS

2FI	-	Two-Factor Interaction
ANOVA	-	Analysis of variance
ASTM	-	American Standard Test Method
BET	-	Brunauer-Emmett-Teller
CFRP	-	Carbon fiber reinforced polymer
CMC	-	Ceramic matrix composite
CNT	-	Carbon nanotube
COF	-	Coefficient of friction
CTE	-	Coefficient of thermal expansion
DFT	-	Density functional theory
DGEBA	-	Diglycidyl ether of bisphenol A
EC	-	European commission
EHT	-	Electrical high tension
EoL	-	End of Life
EP	-	Epoxy
EP/rCF-AR	-	Epoxy reinforced as-received recycled carbon fiber
EP/rCF-T	-	Epoxy reinforced treated recycled carbon fiber
EP/rCF-T/CNT	-	Epoxy reinforced treated recycled carbon fiber and carbon nanotube
FBP	-	Fluidised Bed Process

FESEM	-	Field Emission Scanning Electron Microscope
FRP	-	Fiber reinforced polymer
MMC	-	Metal matrix composite
MWCNT	-	Multi-walled carbon nanotube
PMC	-	Polymer matrix composite
rCF	-	Recycled carbon fiber
rCF-AR	-	As-received recycled carbon fiber
rCF-T	-	Treated recycled carbon fiber
RSM	-	Response surface methodology
SCF	-	Short carbon fiber
SE	-	Secondary electron image mode
SEM	-	Scanning Electron Microscope
SiC	-	Silicon carbide
SiO_2	-	Silicon dioxide
TiO_2	-	Titanium dioxide

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1. Law, M.L., Qumrul, A., Hairul, E.A.M, Noraiham, M. and Sivaraos (2015). Wear Behaviour of Cryogenic Treated Recycled Carbon Fibers Filled Epoxy Composite. *Applied Mechanics and Materials*, 761, pp.489-493.

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CHAPTER 1

INTRODUCTION

This chapter contains the background of research, problem statement, objectives as well as scope of the study.

1.1 Background Study

In the recent years, reinforcement of carbon fibers for thermoset polymer materials has formed a very important class of tribo-engineering materials in nonlubricated condition. This is due to the composite's low density as well as good mechanical and tribological properties that are comparable to or even better than that of many conventional materials. As carbon fiber is a valuable commodity, owing to its specific strength, specific modulus and thermo-physical properties (Akonda et al., 2012), it would make more sense to reuse the recycled carbon fiber from the growing amount of non-degradability of carbon fiber reinforced polymer (CFRP) composite waste material.

Grinding up the end-of-life waste material and sending it to landfill and/or for incinerating has been commonly practiced as it has little appeal to industries due to its degraded properties and the existence of epoxy thermosets on the surface of the recycled carbon fibers. Therefore, to produce valuable product of recycled carbon fibers, surface treatment via cyclic cryogenic treatment is introduced to shell off the epoxy layer as well as to increase the surface roughness of the fibers for better mechanical interlocking

between the fibers and the matrix (Zhang et al., 2004a). The shelling off of epoxy remnants is crucial as it would deteriorate the performance of composite due to poor bonding with the polymer matrix. The fact is that the properties of a composite are influenced by the interfacial adhesion between fibers and matrix. Favorable interfacial adhesion can efficiently transfer load from one fiber to another efficiently through the matrix, resulting in stronger composites which plays a key role in determining the mechanical properties of the composites (Dai et al., 2011; Zhang et al., 2004a).

In addition, to facilitate more severe applications arising from the wear situations, integrating nano-sized fillers is a route to develop high performance composite materials which cannot be achieved by using single filler alone (Chang and Friedrich, 2010). Nanoscale material exhibits extremely high surface area that is able to create a great amount of interphase in composite to build a strong interaction between the fillers and the matrix (Lin et al., 2012). Guo et al. (2009) has proven that nanometer fillers have demonstrated the ability in reducing the friction and enhancing the wear resistance of polymer composites.

Nevertheless, the properties of a composite are also generally dependent on the proper processing technique of composite. Advancement of technology development namely, vacuum casting technique has enabled the production of bubble-free, good dimensional accuracy and good surface quality product (Mais, 1991). In addition, the method enabled rapid production and production in larger quantities.

Composite materials with superior quality and long life span are desired by industries such as for sports equipment. It is therefore of importance to improve the waste management of the composite's waste in order to ensure the sustainability of the material at the end services.

1.2 Problem Statement

The major barriers to the widespread of carbon fiber usage are their high cost and the uncertainty about whether they can be recycled when the composite products reach the end of their useful lives. Furthermore, due to the growing volume of carbon fiber composites' usage, the increasing amount of waste generated from the end-of-life components and manufacturing scrap has become an issue as the waste products are not biodegradable. An example is carbon fiber reinforced polymer (CFRP) composites' woven prepreg waste; materials impregnated with epoxy resin. The challenge is to produce valuable product of mechanically recycled carbon fiber product from the waste and reuse the recycled carbon fibers as reinforcements in new composite material. This study is significant as it is capable of reducing the CFRPs waste in the landfill that could be harmful to the human health and the environment as well as encouraging the usage of recycled carbon fibers in industries instead of using the virgin carbon fibers.